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The effects of sputtering conditions on the structure and magnetic properties of $Nd_2(Fe_{0.9}Co_{0.1})_{14}B$ films and multilayers are reported. The samples were sputtered onto temperature-controlled mica and Ta substrates, at temperatures between 20 and 620 °C. X-ray diffraction results indicate that films deposited directly onto 500–600 °C substrates with Fe buffer layers are nearly single-phase materials with the *c* axis of the grains largely perpendicular to the film plane. Room-temperature magnetization measurements to 17.4 kOe indicate that these films have strong perpendicular anisotropy with coercivities in excess of 10 kOe and magnetizations greater than 100 emu/g. Results for $Nd_2(Fe_{0.9}Co_{0.1})_{14}B$/Fe multilayers show that films can be produced with either in-plane or perpendicular anisotropy depending on the layer thicknesses. The Fe multilayers were found to have coercivities similar to those for the single layer films. Multilayers made with Ag were found to have significant interdiffusion and secondary phases.

INTRODUCTION

Recently we demonstrated that films having the tetragonal $Nd_2Fe_{14}B$ (2:14:1) structure and controllable anisotropy can be prepared by sputtering from a single Co-doped target.[1] Our primary motivations for this work are as follows: (a) the control of the growth of single-phase films and the control of their magnetic anisotropy; (b) the study of solid interfaces of 2:14:1 material with magnetic and nonmagnetic metals; (c) the investigation of magnetic coercivity due to domain wall pinning at planar defects[2,3]; and (d) the potential applications of these films in integrated microelectronic and micromechanical devices.

Other recent work on thin-film rare-earth transition-metal permanent magnets has been reported by Cadieu.[4]

In this paper we report the effects of substrate temperature, substrate material, and buffer layers on the growth of the $Nd_2(Fe_{0.9}Co_{0.1})_{14}B$ phase and the magnetic properties of the films. Some magnetic and structural results for a series of $Nd_2(Fe_{0.9}Co_{0.1})_{14}B$ multilayers with Fe and Ag are also reported.

EXPERIMENTAL METHODS

The samples were prepared in a multiple-gun sputtering system with a temperature-controlled substrate holder. The holder was precisely positioned over the appropriate gun by a computer-controlled stepping motor. The $Nd_{17}(Fe_{0.9}Co_{0.1})_{76}B_7$ (NFB) target was placed in the rf gun and was sputtered at about 6 Å/s until the NFB film was 1 μm thick. For the multilayers with Ag and Fe, the Ag was dc sputtered at 5 Å/s and the Fe was sputtered in a modified dc gun at 1 Å/s. The buffer layers were 100–500 Å thick and the individual layer thicknesses in the multilayers were 50–200 Å for the NFB and 5–200 Å for the Fe or Ag with the total multilayer thickness being 1 μm.

The thickness of each film was checked by comparing the weight of the sample with the weight predicted from the sputtering rates and the modulation length of the Ag multilayers was checked by low angle x-ray diffraction. These results generally agreed within an experimental error of about 5%.

The substrates used for this study were mica and Ta. The films adhered well when deposited on room-temperature substrates; however, the films deposited on 300–600 °C mica substrates were apparently strained because the films would easily peel from the substrates unless handled with great care. For this reason the films were lifted from the mica with cellophane tape prior to the magnetization measurements.

A magnetic field of about 1.3 kOe was applied parallel to the film plane during the production of the multilayers.

Structural information was obtained with a Rigaku diffractometer with large-angle and small-angle goniometers and the magnetic data were obtained with a room-temperature vibrating-sample magnetometer (VSM) with a maximum applied field of 17.4 kOe and a low-temperature VSM with a maximum field of 80 kOe.

RESULTS AND DISCUSSION

Our x-ray results indicate that the 2:14:1 structure is best formed when the films are sputtered directly onto a 500–600 °C mica or Ta substrate, but films deposited at any temperature and then annealed at 600 °C for 25 min consist of a rhombic Nd_2Fe_{17} (2:17) type of phase in addition to the 2:14:1 phase. A possible explanation for this behavior is that when the films are annealed the Nd has time to react with any contaminants in the film, reducing the amount of Nd available to form the 2:14:1 phase to the extent that the formation of 2:17 is favored. Additional support for this idea comes from our attempts to make NFB/Ag multilayers; samples deposited onto room-temperature substrates had large-angle x-ray patterns consistent with amorphous NFB and crystalline Ag, and samples that were annealed or deposited directly onto 600 °C substrates had patterns consis-

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tent with the existence of 2:17 and cubic AgNd as the major phases.

The NFB films and multilayers discussed below were deposited directly onto 500–600 °C substrates. A typical x-ray diffraction pattern for samples deposited on mica is shown in Fig. 1(a). Most of the lines in this figure can be indexed as 2:14:1 lines with the relative intensities indicating the *c* axes of the grains are largely perpendicular to the film plane; however, this figure also shows there is a significant amount of NdO present. Figure 1(b) shows the effect of a 500-Å Fe buffer layer between the substrate and NFB film. The NdO lines diminish as the buffer layer is made larger and disappear completely if a Ta substrate is used. This indicates that the NdO is the result of a reaction with the mica substrate. In the case of the Ta substrate additional lines from an unidentified phase are present. These lines are probably also due to a substrate interaction with the NFB.

The large angle diffraction patterns for the NFB/Fe, Fig. 1(c), multilayers are similar to those for the NFB films with the exception that small amounts of Nd are detectable in some of the samples and the relative intensities indicate that the *c* axes of the 2:14:1 grains begin to tip into the film plane. Studies are underway to determine if this tip is due to the external field applied during sputtering.

Magnetization measurements on the NFB films indicate, that all of samples deposited on 500 °C substrates have perpendicular anisotropy. In the NFB/Fe multilayers perpendicular anisotropy is observed for Fe layer thicknesses ranging from 0 to about 20 Å and NFB thicknesses greater than 100 Å. The x-ray data indicate that the samples that exhibit perpendicular anisotropy also have the *c* axes perpendicular to the film plane and the samples with in-plane anisotropy have some grains with *c* axes in the plane.

The heat-treated Ag multilayers contained no detectable 2:14:1 or Ag phase, but had coercivities of about 3 kOe with in-plane anisotropy for NFB layers larger than 100 Å and Ag layers smaller than 12 Å.

Hysteresis loops for several samples are shown in Fig. 2.

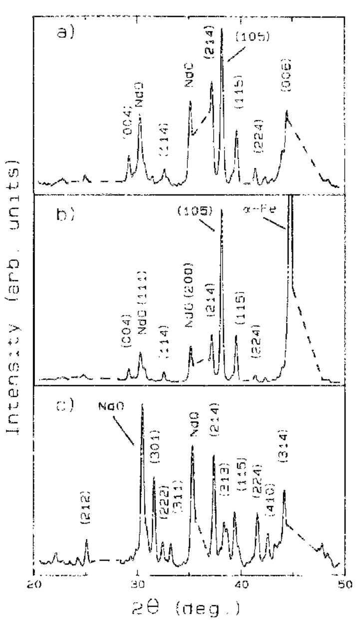

FIG. 1. Selected x-ray diffraction patterns for samples deposited on mica. The mica peaks have been removed for clarity and important peaks have been labeled. (a) is the pattern for an NFB film deposited onto a 500 °C substrate with no buffer layer. (b) is an NFB sample deposited onto a 500 °C substrate with a 500-Å Fe buffer layer. (c) is an NFB(200 Å)/Fe(10 Å) multilayer deposited onto 550 °C mica. This pattern indicates that the *c* axis of some of the grains are in-plane.

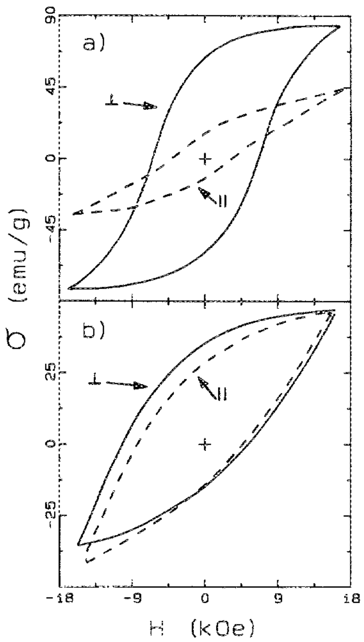

FIG. 2. Hysteresis loops for (a) an NFB film deposited onto a 500 °C Ta substrate with a 200-Å Fe buffer layer and (b) an NFB(200 Å)/Fe(5 Å) multilayer deposited onto a 550 °C mica substrate.

The coercivities of the NFB films and multilayers are typically between 5 and 10 kOe at room temperature and a maximum applied field of 17.4 kOe. Under these conditions the magnetizations of these materials range from 50 to 100 emu/g, but it should be noted that these measurements are from minor loops.

Magnetization measurements up to 80 kOe between 20 and 200 K on NFB(200 Å)/Fe(5 Å) show a monotonic increase in the magnetization from 50 emu/g at 200 K to 80 emu/g at 20 K. This increase may be due to a second phase ordering at low temperature.

In order to study the mechanism responsible for magnetic reversal in these films and multilayers we have measured coercivity versus the maximum applied field for several samples. The results for an NFB(200 Å)/Fe(5 Å) multilayer are shown in Fig. 3. These results are typical of both films and multilayers and suggest that in the film plane the magnetic reversal is controlled by domain wall pinning.[5] In the case of the magnetic field in the perpendicular direction the field dependence of H_c does not fit any simple mechanism. Further studies of this field dependence at higher fields and lower temperatures are under way.

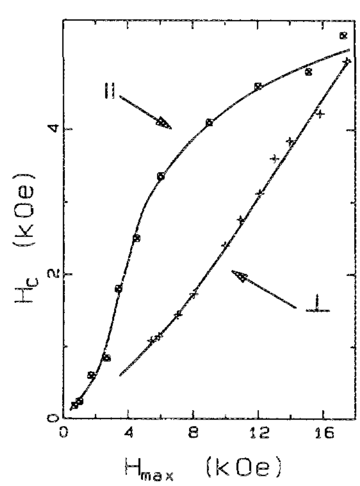

FIG. 3. H_c vs H_{max} for NFB(200 Å)/Fe(5 Å) deposited onto 550 °C Ta. This figure indicates that domain wall pinning controls the magnetization reversal in the plane of these films.

CONCLUSIONS

We have found that sputtered films with the tetragonal 2:14:1 structure can be produced by sputtering onto 500–600 °C mica or Ta substrates. These films contain secondary phases that appear to be due to strong interactions between the films and the substrates. These interactions can be reduced by depositing several hundred angstroms of Fe as a buffer layer between the substrates and NFB film.

The room-temperature coercivities of these samples were found to be between 5 and 10 kOe with magnetizations at 17.4 kOe of 50–100 emu/g. The coercivities of the multilayers decreased with increasing Fe layer thickness.

The mechanism controlling magnetic reversal in the film plane appears to be domain wall pinning but further studies including Lorentz electron microscopy are required to further elucidate this phenomenon.

ACKNOWLEDGMENTS

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